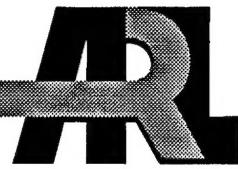


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Effect of a Warm, Moist Environment on JA2 Gun Propellant

by R. Pesce-Rodriguez, R. Leffers,
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ARL-TR-1529

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Effect of a Warm, Moist Environment on JA2 Gun Propellant

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Abstract

Grains and sticks of JA2 propellant downloaded from a 120-mm tank round that had been subjected to conditioning at high temperature and humidity were observed to be discolored and covered with an unknown liquid. Analysis of the propellant indicated a decrease in the nominal diethylene glycol dinitrate (DEGDN) plasticizer content. The nitroglycerine (NG) content was not observed to deviate significantly from its nominal level. The cause of the discoloration of the propellant was traced to its high moisture content. The liquid covering the material was determined to be a mixture of water and DEGDN. In follow-up experiments, it was determined that JA2 propellant can absorb an excess of 13 wt-% moisture. It was also learned that graphite glaze can reduce the tendency of a grain to absorb moisture for grains immersed in water. When conditioned in a high-humidity environment, graphite glaze appeared to have no effect on the level of moisture absorbance. It is suspected that graphite serves to plug pores on the surface of the propellant and thereby inhibit, but not prevent, moisture absorption.

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1. Introduction

A solventless desorption gas chromatography mass spectroscopy (GC-MS) method for determination of moisture in solid gun propellants was developed in 1995 [1]. There are several traditional methods that might otherwise be used to determine moisture content of solid propellants (e.g., gravimetric techniques that involve heating the propellant and monitoring weight loss, and MIL-STD-2668 [2] or JANNAF 523.1 [3], which involve extracting water with dry solvents and then analyzing the extract by liquid or GC). The new desorption-GC-MS method was developed to meet the urgent request for analysis of JA2 samples suspected to have been exposed to excessive moisture. Conventional extraction methods could not be employed because there was neither time to dry the required solvents (MIL-STD-2668 recommends that solvents remain over molecular sieves for a minimum of two days before use) nor the local environmental conditions to keep the solvents dry (due to high, ambient humidity and the absence of adequate air conditioning). To meet the suspense for the required analyses, it was decided that an alternate, solvent-free technique be developed. This was successfully accomplished and yielded results in a relatively short period of time (instrument calibration plus approximately 30 sample runs in 10 hr). In addition, the method did not require the use of volatile organic compounds (VOCs) and consumed only small amounts of propellant. The main disadvantages of the method are that multiple analyses are required to assure representative results (since the test requires a very small sample size), and specialized instrumentation (e.g., a device in which materials may be desorbed from the propellant and then transferred directly into a GC-MS) is required.

Numerous samples of JA2 propellant have been analyzed using this convenient method. In most cases, the propellants were found to be within specifications. In one case, JA2 propellant that had been downloaded from a tank round that had been subjected to extreme temperature cycling at 95% relative humidity was analyzed and found to be several weight-percent below specifications with respect to plasticizer content and several weight-percent above specifications with respect to moisture content. The color of the samples was yellow/green, as opposed to the usual black/green color of "normal" JA2 propellant. When the yellow/green propellant was allowed to dry in a

desiccator, it was observed to turn black/green. A sample of the dried, black/green propellant, which was subsequently placed in a warm, high-moisture environment, was observed to absorb moisture and revert to the previous yellow/green color. "Standard" samples of JA2 propellant placed in the same environment underwent the same color change on absorption of moisture.

While the information regarding plasticizer content was of interest, it was perhaps of greater interest that JA2 could absorb enough water to result in a color change. The level of moisture found in the samples was well above that previously thought possible for JA2 propellant (i.e., ~1 wt-%). Given the significant effect that moisture can have on gun performance, it was decided that the issue of moisture absorption under "extreme" conditions deserved further attention. This report presents results of analyses of the downloaded propellant, as well as propellants conditioned in our laboratory. The goal of analyzing the latter was to provide a better understanding of the upper limit of moisture absorption by JA2, as well as the effect of graphite-glazing on moisture absorption. Also discussed is a method for instrument calibration that is an improvement over that described in Pesce-Rodriguez [1].

2. Experimental

2.1 Samples. Samples downloaded from a M829A2 120-mm armor-piercing, fin-stabilized, discarding sabot with tracer (APFSDS-T) tank round, including JA2 propellant, combustible cartridge case, adapter, propellant bag, and granular propellant bag, were made available by the Aberdeen Test Center (ATC) for analysis. The round had previously been subjected to temperature-cycling at 95% relative humidity for 28 days, following the outlined program as follows.

Step 1 - Propellant held at 21° C for 1 hr.

Step 2 - Temperature reduced to -54° C over the course of 2 hr.

Step 3 - Temperature held at -54° C for 5 hr.

Step 4 - Temperature raised to 71° C over the course of 3 hr.

Step 5 - Temperature held at 71° C for 13 hr.

Step 6 - Temperature reduced to -54° C over the course of 3 hr.

Step 7 - Repeat steps 3–6 until a total of 28 days has elapsed.

Note that the tank rounds were not in sealed canisters when tested; the only protection for the components within the round was the combustible case.

Samples conditioned in the lab were either hexagonal grains with 19 perforations and graphite glaze (1.1-cm diameter) or partially cut sticks with 19 perforations and no graphite glaze (1.7 mm-diameter). Propellant conditioned at 38% relative humidity (RH) was stored in an autodesiccator. Propellant conditioned at 92%-RH was stored over an 11% sulfuric acid solution. Measurements of percent-RH in both the autodesiccator and 92%-RH chamber were made using a Abbeon certified hygrometer (Model AB167B).

2.2 Instrumentation. Desorption was achieved via a CDS Model 122 Pyroprobe (coil type) connected to a heated interface chamber to the splitless injector of a Hewlett Packard GC-Fourier transform infrared (FTIR)-MS system (Model 5890 GC, Model 5970 MS detector [MSD], and Model 5965 infrared detector [IRD]). The GC column used for plasticizer analysis and initial moisture analyses was a Quadrex capillary column (0.32 mm \times 25 m; 3 μm OV-17 film). Analysis of JA2 samples conditioned in the lab were analyzed using a J&W Scientific capillary column (0.25 mm \times 15 m; 0.25 μm DB5 film). The choice of the column used was based solely on convenience. The GC injector temperature was 200° C . The oven-temperature program used was as follows: 50° C isothermal for 1 min, $70^{\circ}\text{ C}/\text{min}$ to 250° C , 250° C for 4 min (total run time 7.86 min).

Analysis of downloaded JA2 propellant by FTIR microreflectance spectroscopy was performed using a Mattson Polaris FTIR spectrometer interfaced to a Spectra-Tech infrared (IR)-Plan IR microscope with a mercury-cadmium-telluride detector. The microscope was operated in reflectance mode, and aluminum foil was used to obtain a background spectrum. For all spectra, 32 scans were collected with a resolution of 8 cm^{-1} .

2.3 Procedure. Small samples of the solid propellant were cut using a razor blade, weighed, and placed into quartz tubes containing a plug of glass wool. The glass wool was used to prevent propellant from coming out of the tube. (Samples other than the JA2 propellant were not weighed before analysis.) The quartz tube was then placed within the coils of the pyroprobe heating element (see Figure 1), which was subsequently inserted into the pyroprobe interface and screwed into place. At the start of the GC run, a 150° C pulse (20-s duration) was given to the sample via the pyroprobe. It was confirmed that these conditions are sufficient for desorption of all moisture by giving a second pulse to the sample and observing no subsequent moisture desorption.

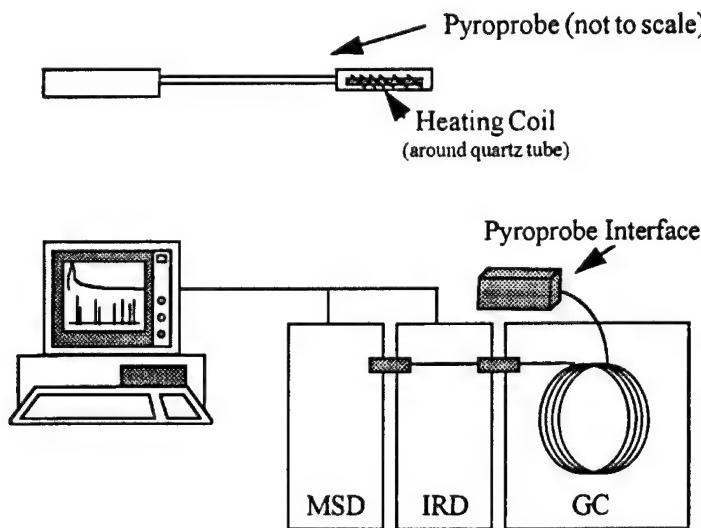


Figure 1. Schematic Representation of Experimental Apparatus.

For preparation of a calibration curve, known masses of potassium sodium tartrate tetrahydrate (PSTT) ($\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) were used. PSTT melts at 70–80° C, loses 3 H_2O at 100° C, and becomes anhydrous at 130–140° C. Desorption of the water of hydration of PSTT was achieved by pulsing at 150° C in a 150° C interface. The PSTT standards were analyzed in the same manner described previously.

Total ion chromatograms (TICs), based on MS response, were collected; selected ion chromatograms (SICs) were also obtained to distinguish between response due to desorbed water and plasticizer (the peaks partially overlap with one another). Integration of the $m/z = 18$ SIC yielded the peak area for water. An example of a TIC and SIC is given in Figure 2. For analysis of plasticizer content, m/z values of 58 and 73 were chose for NG and DEGDN respectively. (For a description of the method for instrument calibration for plasticizer determination, please see Pesce-Rodriguez et al. [4, 5].)

2.4 Instrument Calibration. The moisture calibration curve obtained by analysis of known masses of PSTT is given in Figure 3. (For examples of calibration curves used for determination of plasticizer content, see Pesce-Rodriguez et al. [4, 5].)

Calibration curves obtained using the method described in Pesce-Rodriguez [1] were prepared by desorption of water that had been transferred to a plug of glass wool in a quartz tube by means of a microsyringe. Attempts to use the same method for the current analysis were complicated by the fact that the small quantities of water used tended to evaporate quickly in the low, ambient humidity of the laboratory. (Previous experiments had been performed during the summer when the ambient humidity was relatively high.)

3. Results and Discussion

3.1 Plasticizer Content. Since the focus for this report is on the determination of moisture absorption by JA2 under extreme conditions, plasticizer analysis was performed only on propellant downloaded from the M829A2 rounds. Samples conditioned in the lab were not analyzed for plasticizer content.

Downloaded propellant was found to contain the nominal level of NG. The DEGDN level was found to be between 2 and 6 wt-% less than the nominal value. Variation in the DEGDN levels are suspected to depend on the location in the tank round from which it had been sampled.

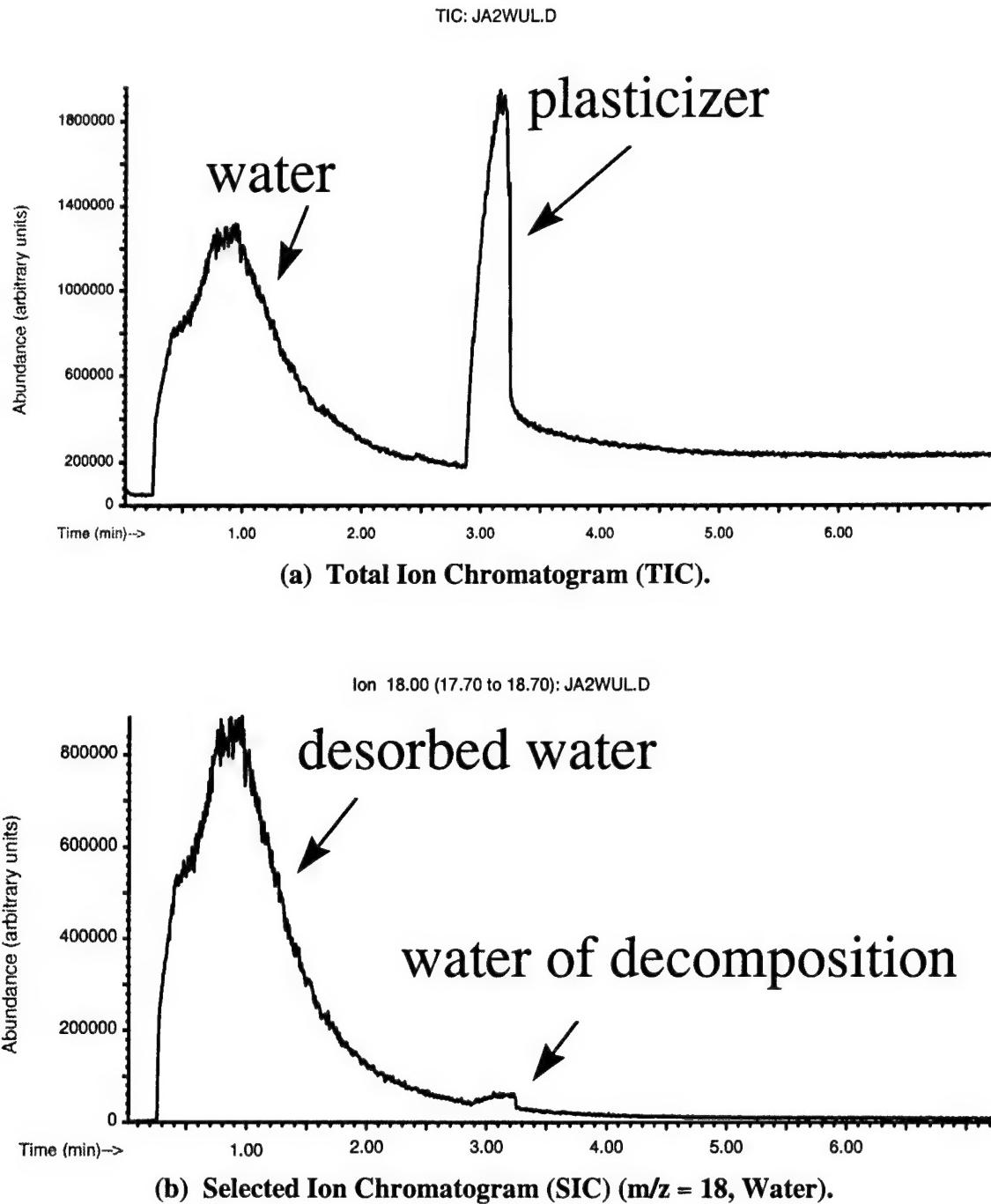


Figure 2. Gas Chromatograms.

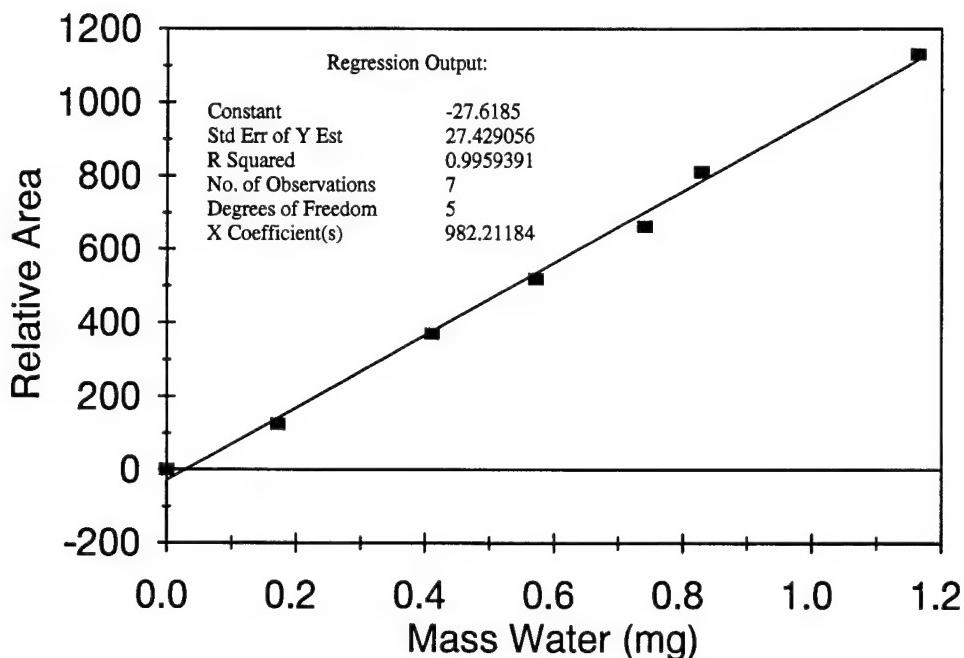


Figure 3. Calibration Curve for Moisture Analysis Generated by Desorption of Water From Potassium Sodium Tartrate Tetrahydrate.

3.2 Stabilizer Content. Unfortunately, no analysis of stabilizer (Arkadit) content was performed; so, it is impossible to comment on the effect of high moisture content on stabilizer content and, therefore, on propellant stability. It is reasonable to assume that absorption of even a small amount of water will increase the likelihood of hydrolysis of nitrate bonds (assuming the presence of trace residual acid from nitration of cellulose). While the same should be true for absorption of large amounts of water (i.e., ~10 wt-%), it is also possible that the large, excess moisture may actually serve to stabilize the propellant, at least in the short term, by acting as a heat sink for exothermic decomposition reactions or other possible ignition sources.

(On a related note: Analysis of World War II munitions recovered from the Baltic Sea in 1993 indicated that sea water markedly slowed the decomposition rate of diphenyl amine [DPA] stabilizer by preventing oxygen from reaching the propellant. The percent nitration of the NC in the

propellant, however, was observed to be significantly decreased. Confirmation of the effect of oxygen on DPA decomposition was confirmed by subsequent control experiments [6].)

3.3 Nitrocellulose Analysis. Comparison of the IR spectra of downloaded JA2 with that of a standard JA2 sample indicate that the downloaded samples had not undergone a detectable degree of denitration as evidenced by the absence of carbonyl peaks (which absorb near $1,700\text{ cm}^{-1}$) in their spectrum (see Figure 4). The presence of carbonyl peaks would have been indicative of the loss of nitrate functional groups with the resultant formation of aldehyde functional groups. No measurements of the molecular weight of the NC were made.

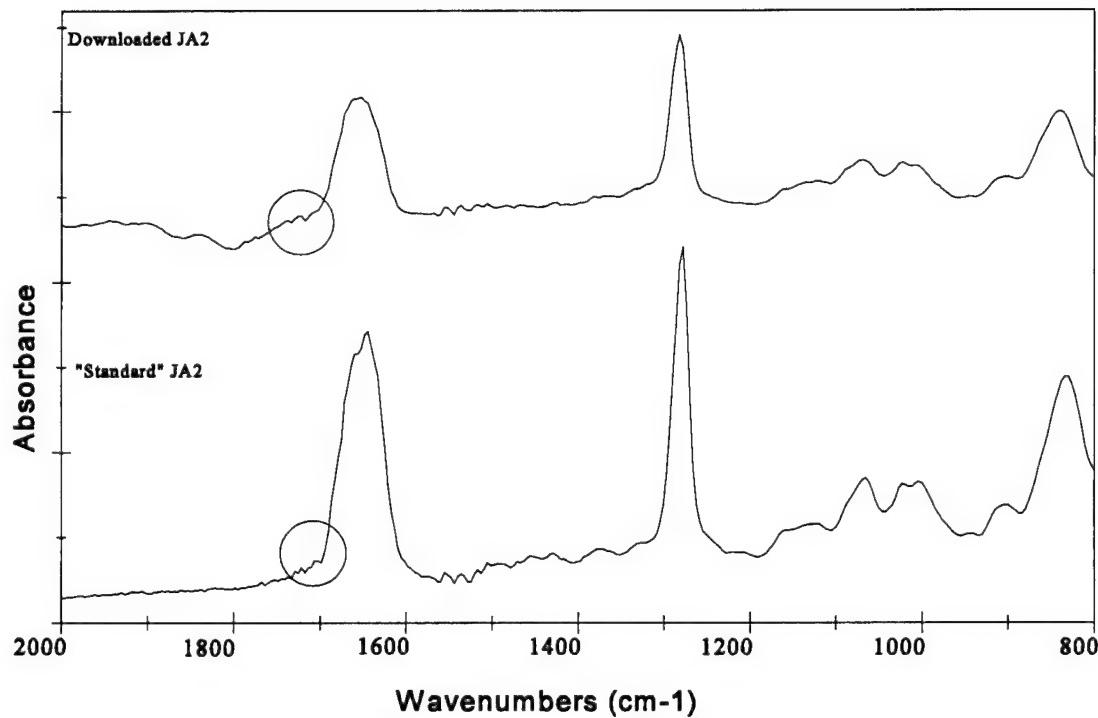


Figure 4. FTIR Spectra of Standard and Downloaded JA2 Propellant. (Circles indicate the region of the spectrum in which carbonyl functional groups would absorb if they were present in the samples.)

3.4 Identity of Unknown Liquid. Desorption GC-MS analysis of the liquid found on the surfaces of the downloaded propellant and other components of the round revealed that the liquid was a mixture of water and DEGDN. No NG was detected in the liquid.

It is suspected that DEGDN is responsible for most of the water absorption. DEGDN is more likely than NG to form hydrogen bonds with water. Nitrocellulose propellant containing only NG is known to be quite water resistant (and was, at one time, advertised as being "waterproof") [7], whereas neat nitrocellulose is very water absorbent. DEGDN has a much higher vapor pressure than NG (3.6 vs. 0.2 torr at 20° C for DEGDN and NG, respectively) and is known to exude from propellants stored at elevated temperatures.

As a note of interest, [7] there is a sample of 100-yr-old double-base flake propellant (40% NG) at the Hercules Plant in Kenvil, NJ, which was made at the Laflin and Rand Haskell plant (also in NJ) that has been stored under water for most of its lifetime. After being dried in an oven overnight and fired in the small-caliber gun that it was originally designed for, the ballistics have not changed since the day it was made.

3.5 Moisture Content.

3.5.1 Appearance of Wet Propellant. The initial motivation for the chemical characterization of JA2 propellant conditioned under high humidity was a concern of ATC personnel regarding the appearance of the propellant downloaded from M829A2 rounds; the propellant had changed from its initial black/green color to a yellow/green color. It was observed that downloaded propellant returned to its initial black/green color after storage under low humidity conditions. The propellant reverted to the yellow/green color after either soaking in a water bath or being exposed to a warm, moist environment. Photos of JA2 propellant in its "wet" and "dry" conditions are shown in Figure 5. (Note: Three different grain geometries are shown. Difference in size not due to swelling.)

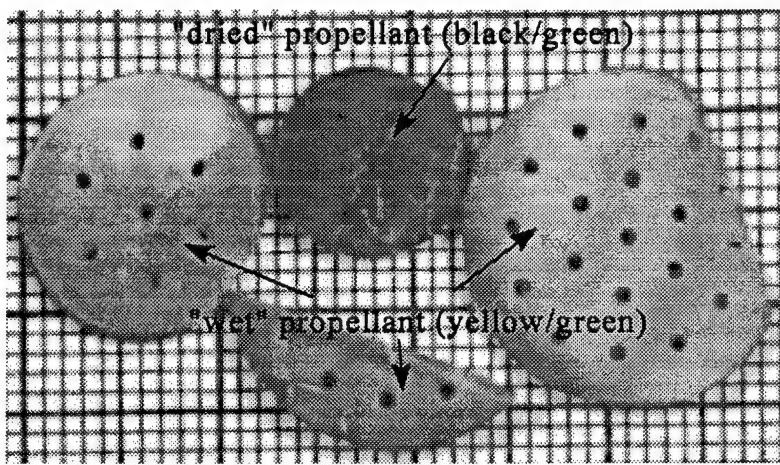


Figure 5. JA2 Propellant Recovered From Downloaded M829A2 Rounds. Dry propellant stored under ambient conditions for several days subsequent to downloading. Wet propellant stored over water vapor (40° C), similar in appearance (color) to material originally downloaded from round. Difference in size not due to swelling.

3.5.2 Desorption-GC-MS Data. The moisture content of the downloaded JA2 propellant provided by the ATC was found to be on the order of 7 wt-%. Based on subsequent analyses of samples prepared in the laboratory, it is suspected that the moisture content of the sample might have been higher had the samples been analyzed immediately after the propellant was downloaded. Table 1 presents the results for moisture analysis of samples conditioned in our laboratory. Note that military specifications for JA2 propellant call for 0.5 ± 0.3 -wt-% moisture.

3.5.3 Comparison of Desorption-GC-MS Data With Karl Fischer Data. Although the desorption-GC-MS measurements of moisture content reported in Table 1 were not compared with side-by-side measurements by a traditional method, the results from propellant conditioned at both 38%-RH and 92%-RH are about what one would expect, based on data obtained using a Karl Fischer method by a laboratory that routinely performs such analyses (i.e., the Gun and Rocket Propellant Lab of the Federal Armed Forces Technical Center for Weapons and Ammunition, Meppen, Germany). In Figure 6, moisture determinations made by the Meppen lab for two "typical" JA2 lots conditioned over the range of 10 %-RH to 95 %-RH are compared with results obtained at the U.S. Army Research Laboratory (ARL) for propellant conditioned at 38%-RH and 92 %-RH.

Table 1. Results for Moisture Analysis of JA2 Propellant

| Sample Identity/Remarks (all samples conditioned for 1 mo) | Peak Area (arbitrary units) | Mass (mg) | Water (wt-%) |
|---|--------------------------------|--------------|-----------------|
| 38%-RH, 22° C; no graphite glaze | 17.1 | 7.900 | 0.35 |
| 38%-RH, 22° C; no graphite glaze | 11.1 | 5.756 | 0.26 |
| 92%-RH, 22° C; no glaze; small piece | 50.3 | 8.848 | 1.14 |
| 92%-RH, 22° C; no glaze; small piece | 32.4 | 5.340 | 1.15 |
| 92%-RH, 22° C; glazed; full grain | 36.5 | 5.923 | 1.19 |
| 92%-RH, 22° C; glazed; full grain | 44.1 | 7.452 | 1.17 |
| water bath; 22° C; no glaze; small piece | 318.4 | 5.020 | 13.71 |
| water bath; 22° C; no glaze; small piece | 376.9 | 6.325 | 12.91 |
| water bath; 22° C; glazed; full grain | 128.6 | 7.290 | 3.73 |
| water bath; 22° C; glazed; full grain | 90.3 | 6.049 | 3.11 |
| water bath; no glaze; full grain ($t = 0.00$ hr at 45%-RH) | 416.4 | 7.991 | 11.3 |
| water bath; no glaze; full grain ($t = 0.48$ hr at 45%-RH) | 151.3 | 7.749 | 4.16 |
| water bath; no glaze; full grain ($t = 0.82$ hr at 45%-RH) | 130.7 | 6.531 | 4.24 |
| water bath; no glaze; full grain ($t = 0.90$ hr at 45%-RH) | 174.3 | 9.926 | 3.87 |
| water bath; no glaze; full grain ($t = 19.0$ hr at 45%-RH) | 25.1 | 11.796 | 0.38 |
| water bath; no glaze; full grain ($t = 19.3$ hr at 45%-RH) | 19.1 | 10.870 | 0.30 |

3.5.4 Effect of Graphite Glaze on Moisture Absorption. As indicated in Table 1, unglazed JA2 propellant can absorb on the order of 13-wt-% moisture when stored under extreme conditions (i.e., in a water bath for 1 mo). Under the same conditions, a glazed grain of JA2 propellant absorbs less than 4-wt-% moisture. It is suspected that the graphite serves to plug pores at the propellant surface and thereby inhibits moisture absorption. A photograph of cross-sectioned glazed and unglazed propellant is shown in Figure 7. Note that the glazed grain (which absorbed only 4-wt-% moisture) is much darker than the unglazed grain (which absorbed 11.3-wt% moisture). As in Figure 5, differences in size are not due to swelling, but different initial grain geometries.

When stored at 92%-RH, both glazed and unglazed propellant absorbed on the order of 1.1-wt-% moisture. In this case, it is suspected that even with many pores plugged with graphite, there are still sufficient sites available for the moisture content to reach 1 wt-%. No color difference was observed between the glazed and unglazed samples stored at 92%-RH.

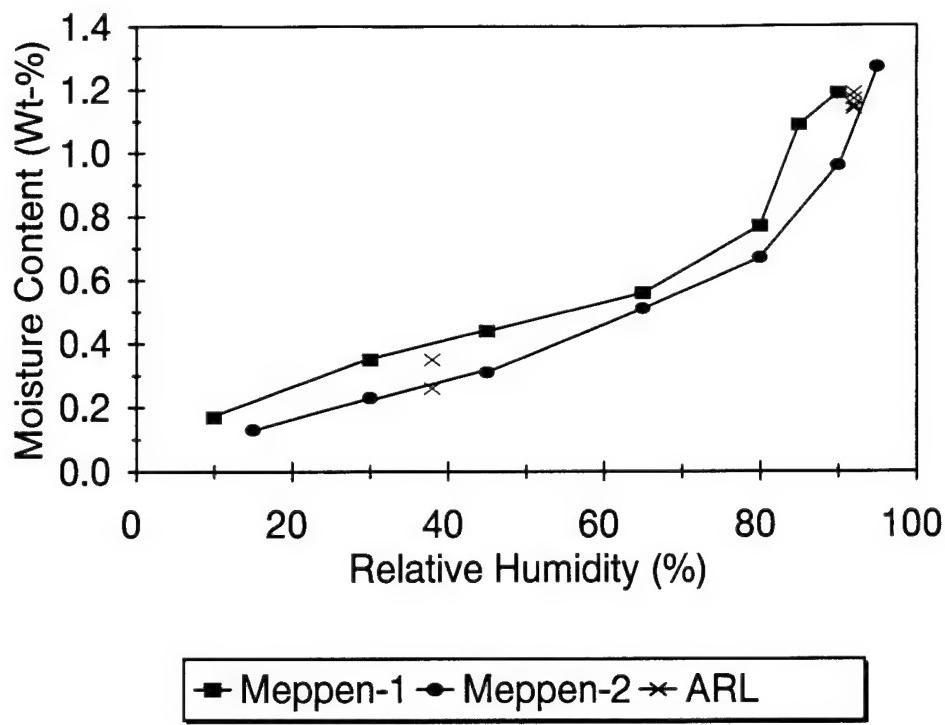


Figure 6. Moisture Content of JA2 Propellant Measured in Meppen (Using a Karl Fischer Method) and by ARL (Using the Desorption-GC-MS Method).

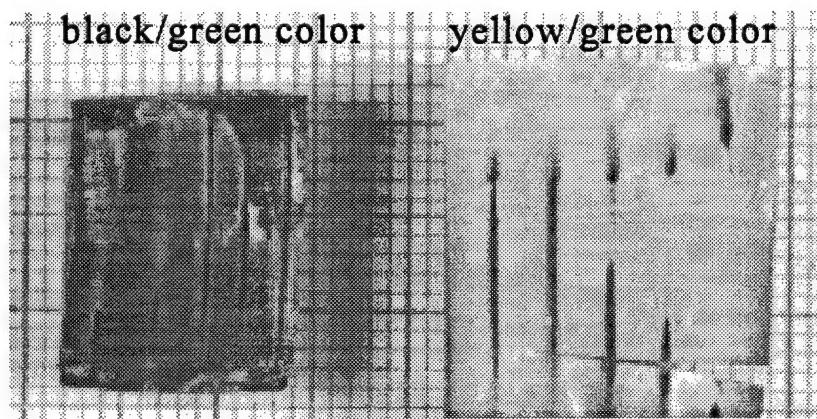


Figure 7. Cross-Sectioned JA2 Propellant That Had Been Stored in a Water Bath for 1 mo. Left: JA2 hexagonal grain (exterior has graphite glaze, 19 perf, and 1.1-cm diameter). Right: JA2 stick (exterior has no graphite glaze, 19 perf, and 1.7-cm diameter). Difference in size not due to swelling.

3.5.5 Estimation of Drying Rate of Wet Propellant. While no formal kinetic study of the drying rate of JA2 propellant was made, it was qualitatively observed (see data in the bottom block of Table 1) that the moisture content of a thin slice of propellant initially containing approximately 11-wt-% moisture drops to approximately 4-wt-% moisture when allowed to dry under ambient conditions (i.e., 43%-RH, 22° C) for just 1 hr. After 20 hr, the moisture content drops to 0.30 wt-% and is probably as low as it will ever be under those ambient conditions.

4. Conclusions

A new technique for calibration of a desorption-GC-MS method was developed. Water desorbed from a hydrated salt (potassium sodium tartrate tetrahydrate) was used to generate a calibration curve that was successfully used to calculate the moisture content of JA2 propellant. The calibration technique was more convenient than one previously described [1].

Conditioning of JA2 propellant (in a fully loaded M829A2 tank round), under conditions of high humidity and temperatures for a period of 28 days, resulted in a depletion of the nominal level of DEGDN plasticizer, but appeared to have no effect on the level of NG plasticizer. No analysis of stabilizer content or NC molecular weight was performed. Analysis of the propellant by microreflectance FTIR spectroscopy suggested that no gross chemical changes had occurred. Changes in coloration of the propellant were ascribed to moisture absorption.

While the "common knowledge" regarding moisture absorption by JA2 was that the propellant does not absorb more than ~1-wt-% water, it has been shown that moisture levels in excess of 13 wt-% are possible.

Color changes (from black/green to yellow/green) are concomitant with absorption of large amounts moisture by JA2 propellant. A return to the original black/green color occurs when the propellant is allowed to dry.

Graphite glaze was found to inhibit absorption of water when propellant is submerged in water, but appears to have little effect when propellant is exposed to high humidity (at room temperature).

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| <p>Grains and sticks of JA2 propellant downloaded from a 120-mm tank round that had been subjected to conditioning at high temperature and humidity were observed to be discolored and covered with an unknown liquid. Analysis of the propellant indicated a decrease in the nominal diethylene glycol dinitrate (DEGDN) plasticizer content. The nitroglycerine (NG) content was not observed to deviate significantly from its nominal level. The cause of the discoloration of the propellant was traced to its high moisture content. The liquid covering the material was determined to be a mixture of water and DEGDN. In follow-up experiments, it was determined that JA2 propellant can absorb an excess of 13 wt-% moisture. It was also learned that graphite glaze can reduce the tendency of a grain to absorb moisture for grains immersed in water. When conditioned in a high-humidity environment, graphite glaze appeared to have no effect on the level of moisture absorbance. It is suspected that graphite serves to plug pores on the surface of the propellant and thereby inhibit, but not prevent, moisture absorption.</p> | | | |
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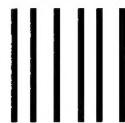
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